

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No. : 10/607,227 Confirmation No. 5896
Applicant : William E. Spindler

Filed : June 26, 2003

Title : CLEANING COMPOUND FOR CLEANING SURFACES
IN A FOOD PROCESSING ENVIRONMENT

TC/A.U. : 1746

Examiner : Bibi Sharidan Carrillo

Atty. Docket No.: NSC0001

Customer No. : 0832

DECLARATION OF WILLIAM E. SPINDLER UNDER 35 C.F.R. §1.132

I, William E. Spindler, declare that:

1. I am the inventor of the invention defined by Claims 37-80 in the present patent application.

2. I am the president of Wayne Chemical Inc., of Fort Wayne, Indiana, a company that I founded in 1969. I earned a Bachelor of Science degree in chemistry from the University of St. Francis in 1965 after attending Indiana University for three years. Over the course of my career, I have created several hundred chemical products and approximately 75 types of chemical application equipment for the food processing industry, and have been listed as an inventor on several U.S. patents.

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3. Independent Claim 37 calls for a method of cleaning and disinfecting a surface or an item of equipment, including the steps of providing a cleaning kit including a first container consisting essentially of a peroxide, and a second container consisting essentially of an alkaline component; and applying the peroxide and the alkaline components of the first and second containers to at least one of the surface and the item of equipment to clean and disinfect the surface or item of equipment.

4. Independent Claim 57 calls for a method of cleaning and disinfecting a surface or an item of equipment, including the step of providing a cleaning composition in dry form, consisting essentially of a peroxide and an alkaline component.

5. Independent Claim 71 calls for a method of cleaning and disinfecting a surface or an item of equipment, including the step of providing a cleaning composition in dry form, the cleaning composition consisting essentially of a peroxide.

6. I have read and studied U.S. Patent No. 5,739,327 to Arbogast et al. ("Arbogast et al. '327"). Arbogast et al. '327 discloses bleaching compositions which include (1) an active oxygen source, such as a peroxide of the type listed at col. 6, lines 45-52, and (2) a nitrile activator of the type set forth at col. 3, line 56 through col. 5, line 40. As discussed at col. 5, lines 30-40, when the peroxide and the nitrile activator are combined in alkaline conditions, they react to form peroxyimidic intermediates. The peroxyimidic intermediates in turn form peroxyimidic acid, which is the bleaching species. As discussed at col. 9, lines 17-42, in one embodiment, a dual delivery system may be provided in which one container includes the nitrile activator, a surfactant, the active oxygen source, and an acidic buffer, and another container includes an alkaline solution.

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7. Based on my review of Arbogast et al. '327 and my knowledge of chemistry, the nitrile activators of the bleaching compositions of Arbogast et al. '327, which are also known in the art as "bleaching activators", react with the active oxygen source in alkaline conditions to form peroxyimidic intermediates which in turn form peroxyimidic acid. The peroxyimidic acid, a peracid, is a potent oxidant and is the actual bleaching species. Attached hereto as **Exhibits 1 and 2** are, respectively, a web page from www.scienceinthebox.com and Lim, S-H et al., *Performance of a new cationic bleach activator on a hydrogen peroxide bleaching system*, (2004), which each discuss the role of bleach activators in reacting with peroxides in alkaline conditions to generate peracids, potent oxidants that are the actual bleaching species.

8. In contrast to the bleaching compositions of Arbogast et al. '327, which include a nitrile activator that reacts with an active oxygen source in alkaline conditions to generate a peracid that is the bleaching species, the cleaning compositions of independent Claims 37, 57, and 71 lack nitrile or other "bleaching activators", but rather clean and disinfect based on the release of oxygen by the peroxide.

9. I have read and studied U.S. Patent No. 5,743,514 to Rees ("Rees '514"). Rees '514 discloses a bleaching solution including (1) a peroxide, such as hydrogen peroxide, (2) an alkaline agent, such as an alkaline metal carbonate, and (3) a lactone of the type set forth at col. 4, line 41 through col. 5, line 6. The disclosure states that "the lactones employed in the inventive solution enhance the bleaching rate of hydrogen peroxide by formation of a peroxyacid of the ring opened lactone in a neutral to alkaline environment" which enhances "the bleaching rate of the inventive solution compared to a similar alkaline solution of hydrogen peroxide without the lactone". Notably, as discussed at col. 5, lines 23-37, the lactone and the peroxide are both more stable under acidic conditions such that the bleaching solution may be provided in two vessels, in which a first vessel includes the lactone and the peroxide, and a second vessel includes at least one alkaline agent (col. 6, lines 11-29).

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10. Based on my review of Rees '514 and my knowledge of chemistry, the lactone in the bleaching solutions of Rees '514 forms a peroxyacid of the ring opened lactone in a neutral to alkaline environment.

11. In contrast to the bleaching solutions of Rees '514, the cleaning composition claimed in independent Claim 37 is based on a peroxide and an alkaline component which do not include a lactone, but rather clean and disinfect based on the release of oxygen by the peroxide.

12. I have read and studied U.S. Patent No. 6,391,840 to Thompson et al. ("Thompson et al. '840"). Thompson et al. '840 discloses bleaching compositions which may include two partial compositions, one of which containing an alkaline pH adjusting compound, and the other containing a peroxide and a bleach activator. Suitable peroxides, or "peroxygen bleach compounds", are set forth at col. 5, lines 39-67, and suitable bleach activator compounds, such as imines and oxaziridines, are set forth at col. 6, line 1 through col. 8, line 54.

13. Based on my review of Thompson et al. '840 and my knowledge of chemistry, similar to Arbogast et al. '327 discussed above, the bleach activators in the bleaching compositions of Thompson et al. '840 react with the peroxide in alkaline conditions to form a peracid which is the actual bleaching species (*See Exhibits 1 and 2*).

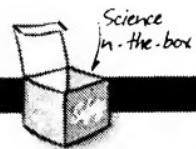
14. In contrast to the bleaching solutions of Thompson et al. '840, the cleaning composition claimed in independent Claim 37 is based on a peroxide and an alkaline component which do not include a bleach activator to generate peracids, but rather clean and disinfect based on the release of oxygen by the peroxide.

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15. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date: 10/25/07

W.E. Spindler
William E. Spindler



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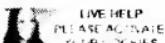
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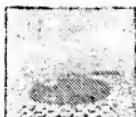
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- Larger stain molecules are broken down into smaller, more water-soluble fragments, which are more easily removed by mechanical action or through the action of other detergent ingredients.

Why use bleach in cleaning products?

Bleach helps the cleaning process by removing and / or decolorizing stains (i.e. whiten or lighten colors). The key mechanism is a chemical reaction that "cuts" the stain molecules in smaller pieces that are more easily removed. Bleach can also act by removing their color so that the stains become invisible. These processes may all work simultaneously on any given organic stain.

Bleach is not only effective on stains but also allows achieving whiteness and dingy cleaning. Dinginess is the overall greyish appearance that white fabrics sometimes develop over time and after many wash cycles.

The first mechanism of bleach action is quite similar to what enzymes do: the larger stain molecules are broken down into smaller, more water-soluble fragments, which are more easily removed by mechanical action or through the action of other detergent ingredients. Unlike enzymes, however, the action mechanism of bleach is a self-destructive one: the bleaching agents disappear in the course of the wash cycle.



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- The bleach reacted with the stain to remove the conjugated double bond system. The colour is gone. This stain becomes invisible

The action of bleach complements that of the surfactants and enzymes in the wash. Some soils that surfactants and enzymes may leave behind, or may only partially remove, are removed by bleach. Also, the fragmenting action of bleach makes the job of surfactants and enzymes during the wash easier.

[◀ back to top](#)**Chemical Functional Definitions**

Bleach is an active molecule that can chemically react with different type of substrates. There are many different kinds of bleaches. When the substrate is a stain, the action of bleach is desirable. When the substrate is a dye in a fabric, the action is not desirable. Chemically speaking, the mechanism of bleaching may be oxidative or reductive; in laundry and cleaning products, oxidative bleaches are used.

Oxidative bleaches are compounds that release hydrogen peroxide (H_2O_2) or singlet oxygen during the wash, or they may be peroxides of organic acids also known as peracids themselves.

Hypochlorite, peroxides, peracids, singlet oxygen are very reactive chemical species; they react with certain parts of organic matter that are responsible for its color. As a result, the organic matter becomes colorless;

hypochlorite/peroxide/peracid/singlet oxygen are used up in the process.

Some of the reactions between bleach and organic matter result in the actual breakage of bonds in the organic matter, thereby releasing smaller molecules of organic matter that are more water soluble.

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Bleach systems that contain active bleaching products and how they work
Hypochlorite, hydrogen peroxide (H_2O_2), singlet oxygen and peracid are the actual, oxidative and so-called bleach actives. They act directly on the stains. The different types of bleaching system have different stain removal/ fabric/color care profile. Formulators choose them based on the benefits they want to achieve.

Several classes of bleaches are known to improve the laundry process:

1. **Sodium hypochlorite (NaClO)** releases highly reactive hypochlorite ions (ClO^-) under alkaline conditions. This is the standard of excellence for stain removal but cannot be used on colored garments. Effective at low temperature and excellent at germ kill. Sodium hypochlorite are not part of a detergent formula but separate product added during the wash process or used directly or after dilution on hard surfaces.
2. **Hydrogen peroxide (H_2O_2)** is one of the most common bleaching agents. The positive aspects of hydrogen peroxide include the fact that it is environmentally friendly (decomposes to O_2 and H_2O), colourless and non-corrosive. To be effective however, hydrogen peroxide requires alkaline conditions and suitably elevated temperatures of about 50°C. Hydrogen peroxide (liquid form) can be used as such in commercial products (i.e. ACE Gentle line up for P&G) or associated with compounds such as borates or carbonates to form solid particles, providing opportunity to formulate hydrogen peroxide in granular detergents.
3. Sodium perborate is one of the most widely used solid peroxygen compounds. However in the last 10 years, mainly as a result of environmental pressures - to reduce boron content of detergents- sodium perborate has slowly been replaced by sodium percarbonate. Sodium percarbonate has a greater rate of dissolution, a more environmentally friendly profile (no release of Boron).
4. Perborate and Percarbonate are usually used in combination with a bleach activator (TAED or Tetra Acetyl Ethylene Diamine) to increase bleaching efficiency at low temperature thanks to the formation of peracetic acid by the reaction of TAED with hydrogen peroxide from percarbonate. In this case, the peracetic acid is the actual bleaching ingredient.
5. **Peracids** work similarly to H_2O_2 but they are more effective than H_2O_2 at low temperatures (below 40°C). Some peracids can be pre-formed and added to the detergent product. However, many peracids are not sufficiently stable to be added to a detergent product. They can be formed during the wash process in presence of hydrogen peroxide and a bleach activator.
6. **Bleach activators** come to the rescue! They are peracid precursors. During the wash, the reaction between H_2O_2 and bleach activators produces peracids; the latter are the actual bleaching agents. A common bleach activator is TAED (Tetra Acetyl Ethylene Diamine). The "-OBS" activators are hydrophobic esters of Oxy Benzene Sulfonate. They work well on dingy cleaning and food stains.
5. **Catalysts** work by making hydrogen peroxide or singlet oxygen more

effective when reacting on stains. They are complex organic molecules with a metallic center. The catalysts are not consumed during the wash process i.e., very small amount can make all the hydrogen peroxide present in the wash process more effective.

6. Photobleaches are also catalysts but they don't work during the wash; instead, they work on the laundry that is hanging on the line outside to dry. Sunlight triggers the formation of singlet oxygen from the air. Singlet oxygen is the actual bleach active.

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Performance of a new cationic bleach activator on a hydrogen peroxide bleaching system

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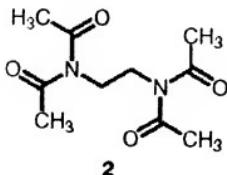
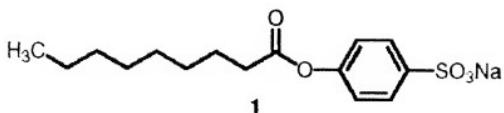
Abstract

The performance of a cationic bleach activator, N-[4-(triethylammoniumethyl)benzoyl]caprolactam chloride, was evaluated in a hot peroxide bleaching process. The effect of time, temperature and the concentrations of hydrogen peroxide and activator on the bleaching of cotton fabric was investigated using a central composite experimental design. Temperature was found to be the most significant parameter. By adding the cationic activator it was possible to achieve a level of whiteness comparable to a typical commercial bleaching system but under relatively mild conditions of time and temperature. As a consequence, chemical damage to the fabric could be reduced. The effect of the cationic bleach activator was compared to that of an anionic activator, nonanoyloxybenzene sulphonate. The cationic activator was superior to the anionic activator in bleaching the fabric under the optimised conditions used in the study.

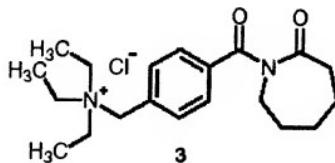
1. Introduction

Cotton fibres contain yellowish impurities which detract from the inherent white colour of the fibre. Unless the fibres are to be dyed deep or dark shades, bleaching is required to remove these natural colorants. Hydrogen peroxide is the most widely used oxidant for this purpose. Bleaching with this material is traditionally carried out under alkaline conditions at temperatures around 95 °C. This involves high energy costs and can also give rise to fibre damage.

Bleach activators are added to promote the oxidation potential of alkaline hydrogen peroxide. These generate peracids, which allow bleaching to be conducted at lower temperatures and for a reduced time, resulting in energy savings and less fabric damage. Among the bleach activators recently developed for incorporation into household laundry detergents, the most common are nonanoyloxybenzene sulphonate (NOBS) (1) and tetra-acetylene diamine (TAED) (2). Following from the increasing awareness of the textile industry to the benefits of activated peroxide bleaching systems, a number of reports have described the use of these two materials [1-5].



Other cationic bleach activators have also been recently reported [6]. These potentially offer increased affinity for the negatively charged surface of cotton in water, as a result helping to minimise hydrolysis in the bleach solution and maximising oxidation at the relevant sites on the substrate. However, no studies have yet been reported on the behaviour of cationic bleach activators in the hot bleaching of textiles. The present study describes the evaluation and optimisation of a novel cationic bleach activator, N-[4-(triethylammoniomethyl)benzoyl]caprolactam chloride (3), using a modification of a typical hot peroxide bleaching system offered by a leading US supplier of textile chemicals.



BY:YANCITY

Experimental

Materials

The fabric used was single jersey circular-knitted greige cotton (175 g/m², 18/1 Ne OE 100%). Tap water was used for the bleaching and washing processes.

The cationic bleach activator 3 was synthesised and purified by the procedure reported previously [6]. NOBS 1 was kindly provided by the Procter and Gamble Company (Cincinnati, USA). The surfactant (Pat-Terge 378 EC), the dispersing agent (Dispersant 2000) and the stabiliser (Stabiliser 128) were kindly provided by Yorkshire Americas, Inc. Other chemicals were purchased from Aldrich Chemical Company (Milwaukee, USA) and Fisher Scientific Company (Suwanee, USA) and

were used without further purification.

Hot bleaching

The bleaching solutions each contained surfactant, dispersing agent, stabiliser, hydrogen peroxide (35%), sodium hydroxide and the bleach activator 3. For all bleedings the quantities of surfactant, dispersing agent, sodium hydroxide and stabiliser were those suggested in a commercial recipe. All bleedings and hot washings were carried out in a laboratory IR dyeing machine (Ahiba Nuance, Datacolor International) at a liquor ratio of 20:1.

After addition of fabric (10 g) to a 250 ml stainless steel beaker containing the bleach solution (200 ml), the temperature was raised to the target bleaching temperature. After the specified bleaching time the bath was then cooled to 50 °C. The fabric was removed and squeezed using a laboratory padder (Warner Mathis AG). It was washed in water (200 ml) at 95°C for 10 min and then cooled to 50 °C. The bleached fabric was finally rinsed thoroughly with tap water and air dried. During both bleaching and hot washing the rate of heating and cooling was 3 °C/min.

Measurement of whiteness

The CIE whiteness index (WI) was calculated for each fabric using AATCC Test Method 110-1995 [7]. Reflectance values were measured on a Datacolor Spectraflash SF 600 Plus-CT using illuminant D65, large area of view, specular included and CIE 1964 supplemental standard 10 observer. Each sample was folded twice to give opacity and the whiteness index was averaged over four separate points on the surface.

Degree of polymerisation of cotton fabrics

The fluidity of the cotton fabric was measured according to AATCC Test Method 82-1996 [7]. The cuen fluidity (F) was converted to the degree of polymerisation (DP) using Eqn 1 [8].

$$DP = 2032 \left(\log_{10} \frac{74.35 + F}{F} \right) - 573 \quad (1)$$

Statistical design of experiments

The bleaching experiments were conducted using central composite design (CCD). The experimental design and statistical analysis were performed using a statistical software, Design-Expert (v. 6.0.8), supplied by Stat-Ease, Inc. (Minneapolis, MN). Four factors potentially affecting bleaching performance were evaluated, namely time, temperature and the concentrations of hydrogen peroxide and activator. The CCD contains five levels for each factor, the coded levels and actual values being shown in Table 1. The parameter to be optimised was the value of CIE WI for the bleached fabrics. The total number of bleaching experiments was 30, including 16 at factorial points, eight at star points and six replications at the centre point. The pattern of the levels of the four factors is shown in Table 2, along with the whiteness values resulting from the individual experiments.

Table 1 Coded and actual levels of the design factors

Factor symbol	Factor	-2	-1	0	+1	+2
X_1	3 ^a (g/200 ml)	0	0.2	0.4	0.6	0.8
X_2	H ₂ O ₂ (g/200 ml)	1.0	1.25	1.5	1.75	2.0
X_3	Time (min)	10	15	20	25	30
X_4	Temp. (°C)	80	87.5	95	102.5	110

a Bleach activator

Table 2 Central composite design for the hot bleaching

No.	X_1	X_2	X_3	X_4	Y (CIE WI)
1	-1	-1	-1	-1	64.82
2	1	-1	-1	-1	67.73
3	-1	1	-1	-1	67.67
4	1	1	-1	-1	70.53
5	-1	-1	1	-1	66.62
6	1	-1	1	-1	69.87
7	-1	1	1	-1	69.12
8	1	1	1	-1	70.01
9	-1	-1	-1	1	69.76
10	1	-1	-1	1	72.47
11	-1	1	-1	1	73.62
12	1	1	-1	1	76.64
13	-1	-1	1	1	73.01
14	1	-1	1	1	74.72
15	-1	1	1	1	75.43
16	1	1	1	1	76.32
17	-2	0	0	0	68.71
18	2	0	0	0	72.10
19	0	-2	0	0	69.39
20	0	2	0	0	73.24
21	0	0	-2	0	69.95
22	0	0	2	0	72.09
23	0	0	0	-2	66.28
24	0	0	0	2	76.57
25	0	0	0	0	71.40
26	0	0	0	0	72.06
27	0	0	0	0	71.83
28	0	0	0	0	71.95
29	0	0	0	0	70.24
30	0	0	0	0	72.04

Results and Discussion

Statistical analysis

The CCD contains five levels for each factor. This number of levels permits the design to generate sufficient data to fit the quadratic model. After analysis of variance (ANOVA) for the quadratic model, including all model terms, those having probability values (p-values) greater than 0.05 were eliminated since they were not statistically significant at the 95% confidence level. The ANOVA data for the significant model terms are given in Table 3 and are based on the use of coded levels for each factor. The model p-value of <0.0001 confirms that the model is significant. The first term, a constant, is referred to as the intercept. The four terms following (X_1 to X_4) are the main factors affecting the response, their values indicating the contribution of each factor [9]. It is seen that temperature had the greatest effect, followed in turn by the concentration of bleach activator (3), the peroxide in describing the nonlinearity of the response. It can be seen that the influence of bleach activator concentration on fabric whiteness is nonlinear. Also, the term $X_2 X_3$ shows that there is a relationship between hydrogen peroxide concentration and time during the process.

Table 3 ANOVA for the quadratic model ($R^2 = 0.9704$)
after exclusion of insignificant terms

Model terms	Estimated coefficient	Standard error	p-value ^a
Constant	71.55	0.14	
X_1	1.25	0.12	<0.0001
X_2	1.21	0.12	<0.0001
X_3	0.71	0.12	<0.0001
X_4	2.72	0.12	<0.0001
X_1^2	-0.47	0.11	0.0003
$X_2 X_3$	-0.38	0.15	0.0177

^a Model p-value = <0.0001

BY: TTF

The lack-of-fit statistic was used to test the adequacy of the model obtained and the p-value was found to improve from 0.7102 to 0.7719 after exclusion of statistically insignificant terms. The lack of fit was not significant, since the p-value of 0.7719 exceeded the 0.05 threshold value. In addition, no abnormality was observed from the diagnoses of residuals. It could therefore be concluded that the model was statistically sound.

The quadratic model predicting CIE WI in terms of actual values of different factors within the scope of the experiments is shown in Eqn 2.

$$\begin{aligned} \text{WI} = & 13.46 + 15.67X_1 + 10.94X_2 + 0.60X_3 \\ & + 0.36X_4 - 11.80X_1^2 - 0.31X_2X_3 \end{aligned} \quad (2)$$

This model was used for prediction of the response (CIEWI) throughout the entire experimental volume evaluated.

Effect of bleaching parameters on whiteness of fabric

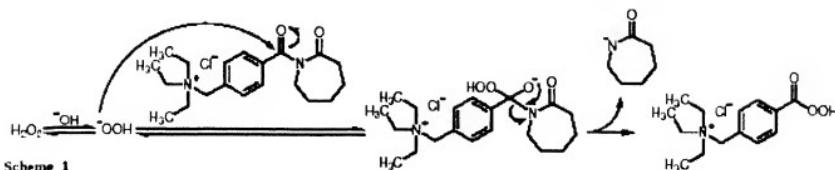
Bleach activation may be defined as the chemical reaction that converts the kinetically less potent bleaching species to one which is kinetically more powerful. As shown in Scheme 1, in the presence of alkali and hydrogen peroxide the bleach activator produces a peracid, which gives a potentially more effective bleaching system than alkaline hydrogen peroxide alone. The peracids generated by cationic bleach activators themselves contain cationic groups, and these potentially increase their affinity for cotton in an aqueous environment. This combination of enhanced affinity and increased oxidation potential leads to improved bleaching performance on cotton. Table 3 ANOVA for the quadratic model ($R^2 = 0.9704$) after exclusion of insignificant terms Model Estimated Standard terms coefficient error p-value The effect of bleaching parameters on the whiteness of bleached cotton is illustrated in Figure 1, in which CIE WI is shown as contour lines and the levels of the parameters not shown for each plot are at their central point (see Table 1 for zero level). It may be observed that within the ranges investigated the whiteness was strongly dependent on bleaching temperature but less so on the time of bleaching.

The performance of a bleaching system has previously been shown to improve as the concentration of hydrogen peroxide or peracid increases [10]. In the present system, which contained both hydrogen peroxide and peracid, the bleaching performance became better as the concentration of either or both components increased. It therefore appears that the two components have an additive effect. It is well known that increased temperature gives a higher rate of bleaching [11]. As shown in Figure 1, the temperature had the largest effect on the whiteness of the cotton. If hydrogen peroxide is used to bleach cotton at room temperature it typically requires at least an overnight dwell time. An increase in temperature and the addition of a bleach activator can improve bleaching performance over a greatly reduced bleaching time [12,13].

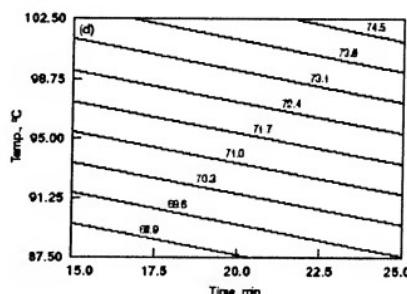
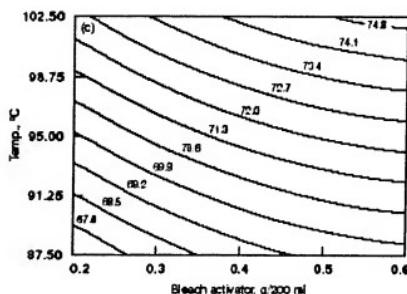
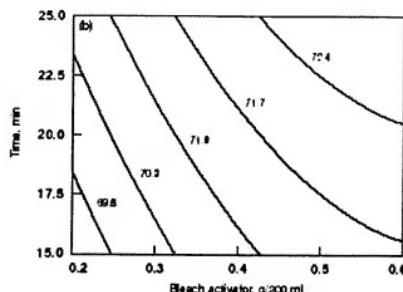
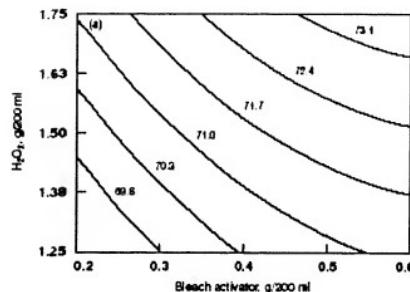
Optimisation of the process

The experimental design software was used to obtain an optimal recipe for the bleaching system. The objective was to establish a recipe which provided a similar whiteness value (75.91) to that given by the original commercial recipe but with a reduced time and temperature, the other factors being kept constant. The components of the commercial and optimised recipes are given in Table 4.

By way of comparison, cotton was bleached using a similar optimised recipe, except the cationic bleach activator 3 was replaced by a molar equivalent quantity of NOBS 1. The CIE WI and DP values for each recipe were calculated and are shown in Table 5, from which it is seen that the measured and predicted whiteness values for the cationic bleach activator were in good agreement. Furthermore, the addition of the cationic bleach activator provided comparable whiteness value to the commercial system in reduced time and at lower temperature. This is beneficial in terms of energy saving and reduced damage to the fabric, as the results in the table show. When a drop of water was placed on fabrics bleached using the cationic bleach activator system and the commercial bleach, respectively, no difference in wettability was observed.



Scheme 1



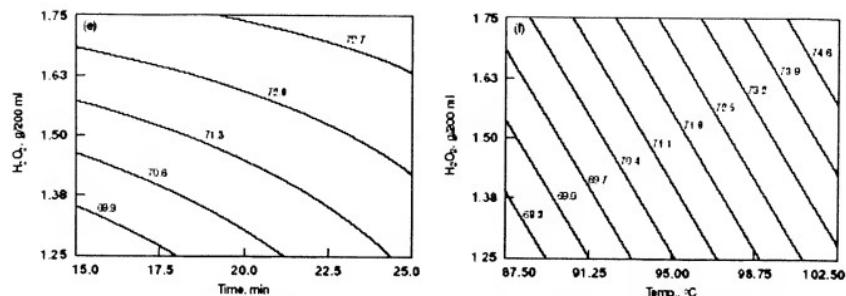


Figure 1 Effect of bleaching parameters on whiteness of cotton; note contour lines represent CIE WI and the levels of parameters not shown for each plot are at their centre point (zero level in Table 1)

Table 4 Commercial and predicted optimised bleach recipes

Recipe ^a	Bleach activator 3 (g/200 ml)	H ₂ O ₂ (g/200 ml)	Time (min)	Temp. (°C)
Commercial		1.71	25.0	110.0
Optimised	0.6	1.71	18.3	102.5

a The quantity of surfactant (0.75 g/l), dispersing agent (1.25 g/l), sodium hydroxide (5.0%, 3 µl) and stabiliser (0.25 g) were identical for both bleach recipes.

Table 5 Whiteness and DP values

System	CIE WI		
	Predicted	Measured ^b	DP ^b
Commercial		75.01	2779
Bleach activator 3	75.91	75.56	2984
NOBS		73.02	3068

a CIE WI before bleaching = -1.24

b DP before bleaching = 3.002

In addition, the cationic bleach activator in this bleaching system produced a higher whiteness level on the fabric than did NOBS, possibly because the cationic bleach activator had a higher affinity for the negatively charged cotton than an anionic bleach activator such as NOBS. Further work will be needed to confirm this.

Conclusions

In the presence of hydrogen peroxide and alkali, bleachactivators generate peracids, which are more potentoxidants. In this study a cationic bleach activator 3 wasemployed to assist a hydrogen peroxide hot bleaching system. It was observed that temperature had the greatestinfluence on the whiteness of cotton fabric, followed inturn by the bleach activator concentration, hydrogen peroxide concentration and time. The predicted and measured whiteness values from the optimised recipe for the cationic bleach activator were in close agreement,confirming the validity of the quadratic model used.

By the addition of the cationic bleach activator 3 to a conventional hot bleaching system, it was possible to obtain a similar level of whiteness at lower temperature and reduced time while maintaining wettability. In regard to fibre damage after bleaching, the cationic bleach activator system gave less chemical damage than the conventional bleaching system, as indicated by the high residual degree of polymerisation. This activator may therefore be beneficial when bleaching fibre blends containing delicate components, for example cotton/wool blends, in which the wool is prone to damage in the hot alkaline conditions.

In this study a typical commercial process has been modified by the simple addition of a novel cationic bleach activator. It is possible that a further reduction in processing time and temperature might be achieved, for example, by the choice of stabiliser and further modification of the structure of the cationic bleach activator.

References

1. J Y Cai, D J Evans and S M Smith, AATCC Rev., 1 (2001) 31.
2. J Wang and N M Washington, AATCC Rev., 2 (2002) 21.
3. A J Mathews, Book of Papers, Proc. AATCC Int. Conf. Exhib., Atlanta, USA (1997) 462.
4. J Y Cai, F J Harrigan and S M Smith, Book of Papers, Proc. AATCC Int. Conf. Exhib., Charlotte, USA (1999) 134.
5. S J Scarborough and A J Mathews, Text. Chem. Colorist Am. Dyestuff Rep., 32 (3) (2000) 33.
6. A D Willey, G S Miracle, K L Kott, M E Burns, G M A Baillely, N Guedira, F E Hardy, L F Taylor and M R Sivik, US5686015 (Procter and Gamble, USA; 1997).
7. AATCC Technical Manual, Vol. 75 (Research Triangle Park: AATCC, 2000).
8. A Bleacher's Handbook (Houston: Solvay Interrox, 1981) 57.
9. R J del Vecchio, Understanding Design of Experiments: A Primer for Technologists (Cincinnati: Hanser/Gardner Publications Inc, 1997).
10. A P James and I S Mackirdy, Chem. Ind. (London, UK) (1990) 641.
11. W S Hickman, in Cellulosics Dyeing, Ed. J Shore (Bradford: SDC, 1995) 117.
12. N G??rsoy, S H Lim, D Hinks and P Hauser, Text. Res. J., in press.

13. N G??rsoy, A El-Shafei, P Hauser and D Hinks, Book of Papers, Proc. AATCC Int. Conf. Exhib., Greenville, USA (2003) 216.

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